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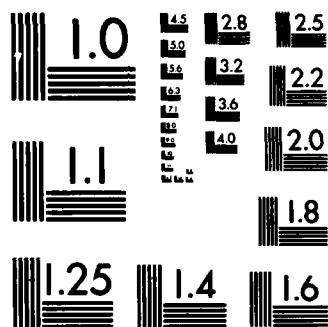
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**CORRELATION OF CHEMICAL
CHARACTERISTICS
WITH FUEL PROPERTIES
BY GAS CHROMATOGRAPHY**

**INTERIM REPORT
AFLRL No. 153**

By

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L.L. Stavinoha**

**U.S. Army Fuels and Lubricants Research Laboratory
Southwest Research Institute
San Antonio, Texas**

Under Contract to

**U.S. Army Mobility Equipment Research
and Development Command
Energy and Water Resources Laboratory
Fort Belvoir, Virginia**

Contract No. DAAK70-82-C-0001

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December 1981

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AFLRL NO. 153	2. GOVT ACCESSION NO. AD-A722708	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) CORRELATION OF CHEMICAL CHARACTERISTICS WITH FUEL PROPERTIES BY GAS CHROMATOGRAPHY		5. TYPE OF REPORT & PERIOD COVERED Interim Report August 1980-December 1981
7. AUTHOR(s) D.L. Present L.L. Stavinocha F.M. Newman		6. PERFORMING ORG. REPORT NUMBER SwRI-6800-124
9. PERFORMING ORGANIZATION NAME AND ADDRESSES U.S. Army Fuels and Lubricants Research Lab Southwest Research Institute P.O. Drawer 28510 San Antonio, TX 78284		8. CONTRACT OR GRANT NUMBER(s) DAAK70-80-C-0001 DAAK70-82-C-0001
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Mobility Equipment Research and Development Command, Energy and Water Resources Laboratory, Ft. Belvoir, VA 22060		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 1L762733AH20EH; WUB01
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE December 1981
		13. NUMBER OF PAGES 43
		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Fuel Properties JP-4 Boiling Point Distribution Analytical Characteristics JP-8 Alternative Fuels Gas Chromatography JP-5 Synthetic Fuels Glass Capillary Gas Chromatography DFM Distillate Fuels		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Standard tests such as those published by ASTM are used to determine a fuel's properties. As new fuels are introduced, it becomes necessary to characterize them by applying available standard test methods and chemical/physical characterization techniques in a process which is costly, time consuming and developmental by its very nature. Because of the technical advances in digital computers and gas chromatography, it has become feasible		

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20. ABSTRACT (Cont'd)

→ to attempt to develop correlations between gas chromatographic data and some chemical/physical properties. These, in turn, may be related to a fuel's performance. Eight test fuels were selected for this preliminary work because of their known stability and chemical/physical properties. A modified ASTM D 2887 boiling point distribution (BPD) method was developed to yield component specific identification in addition to BPD consistent with the conventional D 2887 method. The data from this modified method may be used in correlation equations to automatically calculate Reid Vapor Pressure, ASTM D 86, ASTM D 1160, °API gravity, flash point, viscosity, and freeze point. In addition, the capability to "profile" several chromatograms for direct visual comparison has been developed and added to the system. Other analytical techniques, such as NMR, were evaluated for their possible contribution to this correlation development.

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FOREWORD

The work reported herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (AFLRL), Southwest Research Institute, San Antonio, TX, under Contracts DAAK70-80-C-0001 and DAAK70-82-C-0001 during the period August 1980 through December 1981. The work was funded by the U.S. Army Mobility Equipment Research and Development Command (MERADCOM), Ft. Belvoir, VA. Contracting Officer's representative was Mr. F.W. Schaekel, Fuels and Lubricants Division, Energy and Water Resources Laboratory (DRDME-GL). Project technical monitor was Mr. M.E. LePera, MERADCOM, DRDME-GL.

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ACKNOWLEDGMENT

The authors wish to acknowledge and express their appreciation to the technical staff of the U.S. Army Fuels and Lubricants Research Laboratory for their assistance in the performance of this work. For their assistance with the development and understanding of the computer programs, recognition is made of Mr. E.C. Owens, Manager, Lubricants and Mechanical Systems, USAFLRL, and Mr. D.A. McAfoose, Quality Assurance Systems and Engineering Division, Southwest Research Institute. Appreciation is expressed to Mr. J.W. Pryor and his editorial staff for their assistance in the preparation of this document.

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I. INTRODUCTION

As part of the major thrust within the Alternative and Synthetic Fuels Program, the Department of Defense (DOD) specified in late 1979 a task to "develop more efficient military fuel qualification procedures to effect capacity to react quickly to changes encountered in the petroleum refining industry." The normal time required for qualification of a new fuel for the engine and powerplant accessory systems is approximately 5 to 8 years. As an example, the transition to unleaded gasoline within the Department of the Army took 4 to 5 years.^{(1)*} As shown in Figure 1, the first step in evaluating and qualifying new/modified fuels involves both laboratory characterization and specification testing.

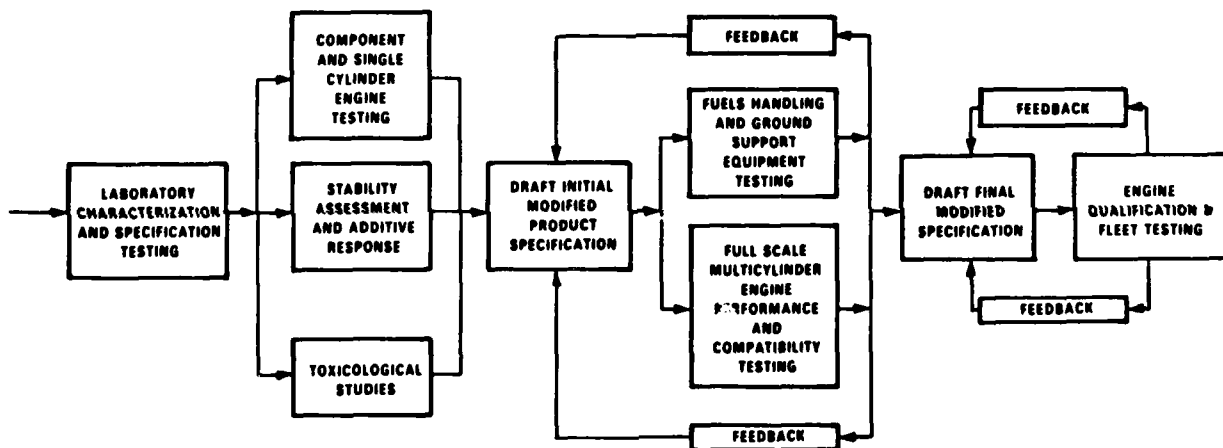


FIGURE 1. PROCESS FOR EVALUATING NEW/SYNTHETIC FUELS

As a part of the development of new accelerated engine/fuel qualification methodology, new instrumental analysis techniques are to be developed which will determine/identify hydrocarbon and nonhydrocarbon constituents in fuels and can be used in the development of more accurate predictive correlations for fuel performance.

* Underscored number in parentheses refer to the list of references at the end of this report.

Fuel characterization is an important consideration for effective spark ignition, compression ignition, and turbine engine fuel utilization. Military mobility equipment depends upon fuels which provide reliable vehicle operation and performance. Military and federal specifications are designed to help control fuel quality for government use by providing the refiner with a guide which aids in producing an acceptable product. Specifications serve this purpose by listing physical and chemical fuel properties provided with maximum and/or minimum data value requirements which a fuel must meet. Table 1 provides a summary of properties and Specification (VV-F-800C) (2) limits for diesel fuels which are used to fuel the majority of Army ground tactical/combat vehicles.

As new fuels are introduced, it becomes necessary to characterize them by applying the available standard test methods and chemical/physical characterization techniques. The standard test methods are both costly and time consuming. When the candidate fuel has been defined by the standard tests, it can then be used in actual engine test stand operation to determine its engine performance characteristics. Engine test stand operation is very costly. At times, the use of a new fuel not previously screened for deleterious properties, can lead to serious engine malfunctions requiring extensive and expensive engine overhaul.

The objectives of this program are (1) to define those fuel properties and characteristics which are the most significant with regard to engine performance, and (2) to attempt to determine those fuel properties and characteristics from a minimum amount of laboratory analytical data through the use of correlation techniques. This report presents the initial results of this program.

TABLE 1. SUMMARY OF PROPERTIES AND SPECIFICATION LIMITS
IN FEDERAL SPECIFICATION VV-F-800C FOR DIESEL FUELS

Properties	Values			
	Grade DF-A	Grade DF-1	Grade DF-2:	
			CONUS	OCONUS
Density, kg/L @15°C	Report	Report	Report	0.815 to 0.860
Flash point, °C min	38	38	52	56 <u>1/</u>
Cloud point, °C max	-51	<u>2/</u>	<u>2/</u>	<u>2/</u>
Pour point, °C max	Report	Report	Report	<u>3/</u>
Kinematic viscosity @40°C (20°C), cSt	1.1 to 2.4	1.3 to 2.9	1.9 to 4.1	(1.8 to 9.5)
Distillation, °C:				
50% evaporated	Report	Report	Report	Report
90% evaporated, max	288	288	338	357
End point, max	300	330	370	370
Residue, vol%, max	3	3	3	3
Carbon residue on 10% bottoms, mass %, max <u>4/</u>	0.10	0.15	0.35	0.20
Sulfur, mass %, max	0.25	0.50	0.50	0.70
Copper strip corrosion, 3 hrs. @ 50°C				
max rating	3	3	3	1
Ash, mass %, max	0.01	0.01	0.01	0.02
Accelerated stability, total insolubles mg/100 mL, max <u>5/</u>	1.5	1.5	1.5	1.5
Neutralization number, TAN, max	0.05	---	---	0.10
Particulate contamination, mg/liter, max	10	10	10	10
Cetane number, min	40	45	45	45

1/ DF-2 intended for entry into the Central European Pipeline System shall have a minimum value of 58°C.

2/ As specified by the procuring activity based on guidance in Appendix A of the Specification. DF-2 for Europe and S. Korea have a maximum limit of minus 13°C.

3/ As specified by the procuring activity. DF-2 for Europe and S. Korea shall have a maximum limit of minus 18°C.

4/ See Appendix B of the Specification. If the fuel contains cetane improvers, the test must be performed on the base fuel blend only.

5/ This requirement is applicable only for military bulk deliveries intended for tactical, OCONUS, or long-term storage (greater than 6 months) applications (i.e., Army depots, etc.).

II. BACKGROUND

Considerable effort has been expended by many researchers to fully identify all the compounds present in petroleum products. Early work by API Project 44 attempted separation and identification by careful distillation and purification. Beginning in the early 1940's, the use of ultraviolet/visible and infrared spectroscopy gave additional insight into the complex structure of fuels.

The advent of gas chromatography (GC)* presented new opportunities and opened new approaches,⁽³⁾ but the many primary compounds with their isomers were not well resolved, and identification of all components continued to be elusive. High-resolution GC with both Support Coated Open Tubular (SCOT) and Wall Coated Open Tubular (WCOT) capillary columns showed that resolution of most compounds in the gasoline range could indeed be accomplished, but identification proved to be an insurmountable task because standard compounds of required purity did not exist for a sufficient number of components to produce detailed results. In addition, the large volume of data involved became a monumental task for reduction and presentation. The refinement and maturation of GC coupled to rapid mass spectrometers (GC/MS) made identification of the hundreds of components appear to be attainable. Employment of other specific detectors such as those which each respond to sulfur, nitrogen, and aromatics further enhanced the possibility of confirming compound identification. The recent use of nuclear magnetic resonance spectrometers as an analytical tool in fuel chemistry has enabled an even closer look into the structure of the many compounds present in a fuel.

Two technical advances have now made this highly desirable goal of complete identification approach a reasonable and attainable level. The widespread use of digital computers as controllers and data processors for analytical instrumentation has increased the capability of the analytical laboratory to handle the mass of data generated by sophisticated instrumental analyses.

* Note: See 1981 Annual Book of ASTM Standards, ASTM E355, the "Recommended Practice for Gas Chromatography Terms and Relationships," part 42, American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.

Also, the improved system stability and performance of the hardware make more reliable data possible.

In 1979, a study was initiated at the U.S. Army Fuels and Lubricants Research Laboratory (AFLRL) to determine the feasibility of combining traditional analytical/chemical instrumentation, engine, and laboratory bench tests into a concise analytical methodology, and from a minimal quantity of compositional and physical data, characterize a substance in terms of its performance as a fuel.

In support of this effort, literature pertaining to physical and chemical methods of characterizing fuels has been reviewed. During this review, it was noted that most physical/chemical fuel properties must be determined directly. However, data for some properties could be calculated using correlative methods. (4,5) A correlative method is an analytical method by which a property can be mathematically determined by using data directly obtained for another property. For example, data from ASTM D 2887 (Boiling Range Distribution of Petroleum Fractions by Gas Chromatography) and ASTM D 3710 (Boiling Range Distribution of Gasoline and Gasoline Fractions by Gas Chromatography) can be used to calculate data for Reid Vapor Pressure and ASTM D 86 (Distillation of Petroleum Products) through mathematical correlation.

As a result of this effort, a report (6) was published providing a reference tabulation of over 100 physical and chemical fuel properties, chemical compounds, and compound classes identified during the literature review along with brief outlines of literature-derived methods for their determination. Methods not treated extensively in this review are developmental methods used primarily in areas of research and development such as fuel lubricity, elastomer compatibility, fuel stability, fleet testing, etc. Many methods of this type are not yet standardized, and various approaches using these methods have been and are being used in fuels and fuels-related research. A great deal of literature exists which discusses these developmental procedures' applications and results in detail. (7-14) Other reports in this general methodology development area at AFLRL have been prepared. (15-17)

III. APPROACH

Another result of the literature survey leading to development of the review discussed in the Introduction (Reference 6), was a directive to evaluate traditional analytical chemical instrument, engine, wet chemical, and bench tests as to their effectiveness in accurately determining physical and chemical fuel properties. Then, by selecting critical fuel-definitive properties, analytical techniques can be developed which will correlate these critical properties with fuel composition and other predetermined physical properties at a high level of confidence by the application of mathematical models.

The literature review afforded various test methods for the determination of over 100 fuel properties calling for utilization of analytical chemical instruments (chromatographs, spectrophotometers, etc.), engines, and bench apparatus. From the review, it appeared that a sophisticated gas chromatographic technique should be explored in a correlative approach to defining fuel physical/chemical properties. The use of external calibration, multicolumns and multidetectors could potentially provide mapping of petroleum and synthetic fuels correlatable to known or definable properties. While this task will not be easily accomplished because of its complexity, the extent of identification will improve with time as the details of the methodology are developed.

IV. DISCUSSION

Several vendors marketing gas chromatographic equipment with the high level of sophistication needed were contacted. After a thorough review and evaluation with consideration toward interfacing with data-handling systems already in-house, it was decided that Hewlett-Packard's 5880A Gas Chromatographic System was the best fit (Figure 2). The 5880A gas chromatograph can contain level 4 BASIC programming, allowing preprogrammed calculation procedures, with access to the GC report information to permit further automatic processing of the GC information. In addition, it has the ability to totally control all GC variables through the use of microprocessors. It can utilize dual capillary columns with the latest innovations in sampling

techniques, and is capable of housing up to four detectors. This multi-detector capability permits the use of selective detectors to minimize downtime for switching between them. The 5880A GC can have Hewlett-Packard's proven cartridge tape unit as a built-in feature. This would provide mass storage for analytical programs, calibration tables, reports, keystroke, and BASIC programs.

The instrumentation purchased incorporates several detectors capable of yielding component and/or element specific information. The capability of detailed composition fingerprinting of the neat fuel as well as the nitrogen and sulfur-containing components and aromatic components will aid in developing correlation of performance properties of experimental and test fuels with the performance properties of known fuels.

Other analytical techniques which would add to the ability to characterize a fuel were developed prior to the installation and operation of the H-P 5880A GC equipment. An infrared technique for the determination of oxygenate concentration of gasoline/oxygenate blends was developed and evaluated. (22) This method proved to be both qualitative and quantitative with a high potential for speed, low cost, and specificity. Additionally, a rapid and inexpensive ultraviolet spectroscopic method for determining the aromaticity of turbine and diesel fuels has been developed. (23) The method yields weight percent ring carbon in substituted benzenes, naphthalenes, and phenanthrenes/anthracenes. The precision and accuracy of the method are good for both standards and fuel blends. The method is currently in use for correlation work for both turbine combustors and diesel engines.

Initially, eight fuels that had previously undergone extensive analyses, and about which much information was available, were selected as a base from which correlative methods might be developed (Table 2). These fuels were derived from a variety of sources, i.e., petroleum, shale, tar sand, and covered turbine and diesel operation. They appeared to be stable over relatively long-term storage conditions, and so could be used over a period of time as an analytical base with a very low probability of changing or degrading.

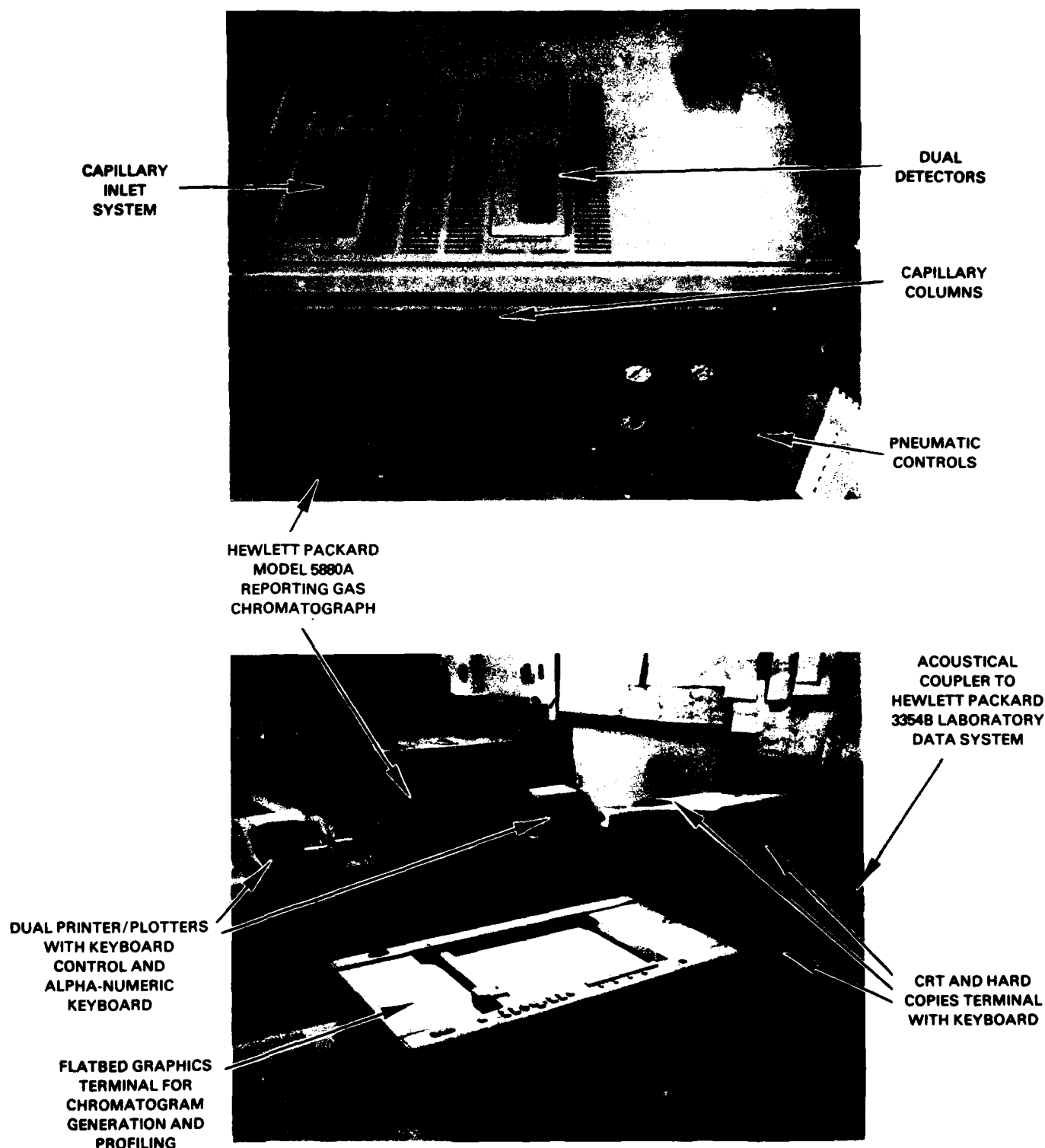


FIGURE 2. GAS CHROMATOGRAPH SYSTEM FOR DEVELOPMENT OF METHODOLOGY FOR FUEL CHARACTERIZATION

TABLE 2. STANDARD BASE TEST FUELS

<u>AFLRL Code Number</u>	<u>Type</u>	<u>Specification</u>	<u>Description</u>
AL-8436-F	JP-5	MIL-T-5624L	Shale-Paraho-II
AL-6354-T	JP-5	MIL-T-5624L	Tarsand
AL-6526-T	JP-5	MIL-T-5624L	Shale
AL-7247-T	JP-5	MIL-T-5624L	Petroleum
AL-9089-SP	JP-8	MIL-T-83133	Shale-Paraho-II
AL-8907-F	JP-8	MIL-T-83133	Petroleum
AL-8437-F	DFM	MIL-F-16884G	Shale-Paraho-II
AL-9847-SP-T	JP-4	MIL-T-5624L	Shale-Geokinetics

High Performance Liquid Chromatography (HPLC) was used to effectively separate the saturates from the olefinic, heteroatomic, and aromatic components of each of the eight test fuels (Figure 3). The separations were clean with only a minimum amount of solvent carryover in some cases.(19)

Each of the eight test fuels and their saturate and aromatic/polar fractions obtained by HPLC fractionation, were analyzed by conventional, packed column GC utilizing flame ionization detectors for ASTM D 2887, "Boiling Range Distribution of Petroleum Fractions by Gas Chromatography" (BPD) (24) (Figures 4, 5, and 6). Table 3 gives the operating parameters for this method using Hewlett-Packard 5711 gas chromatograph. These data were stored on disc in the Hewlett-Packard 3354B/C Laboratory Data System computer used for calculating the boiling point distribution.

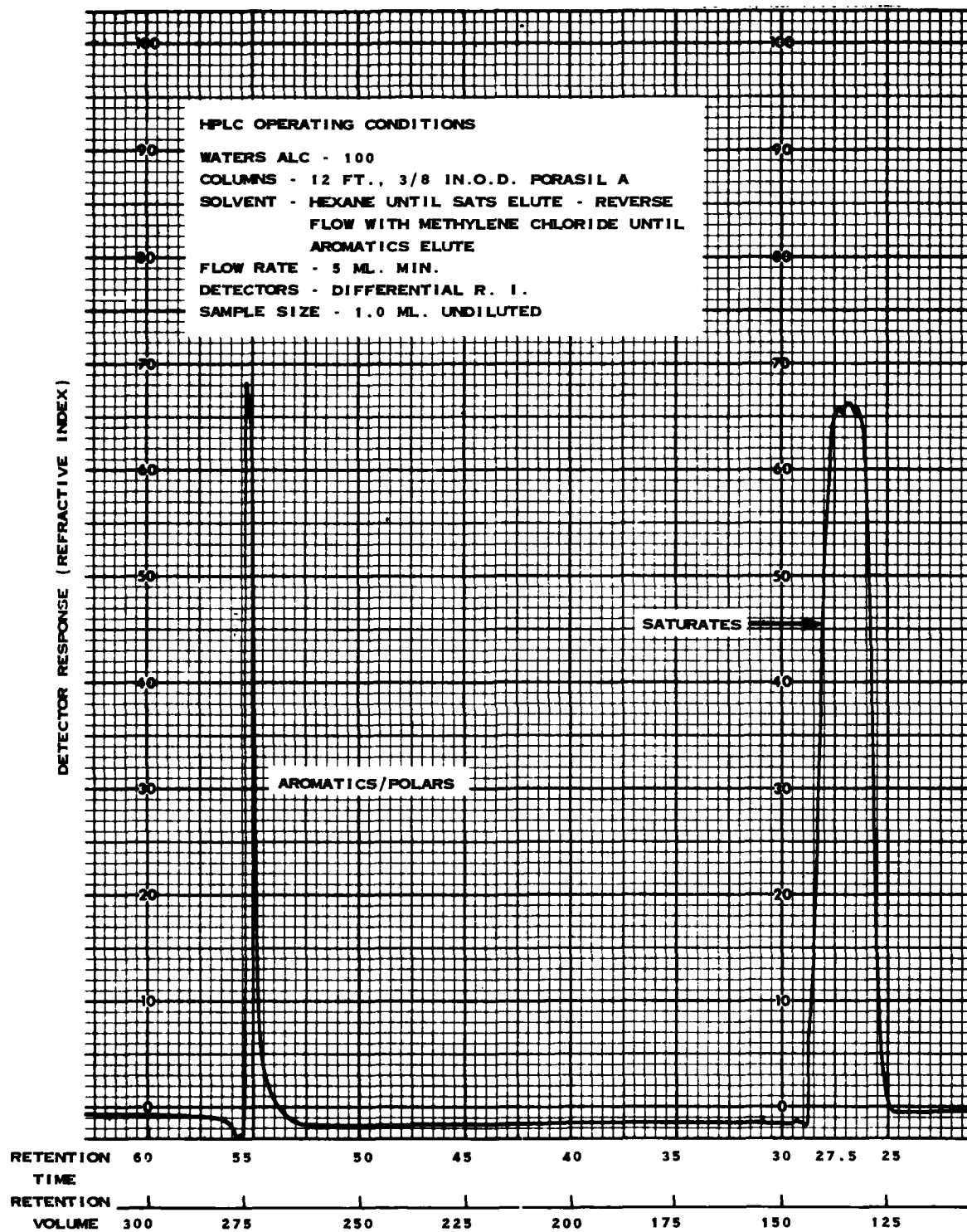
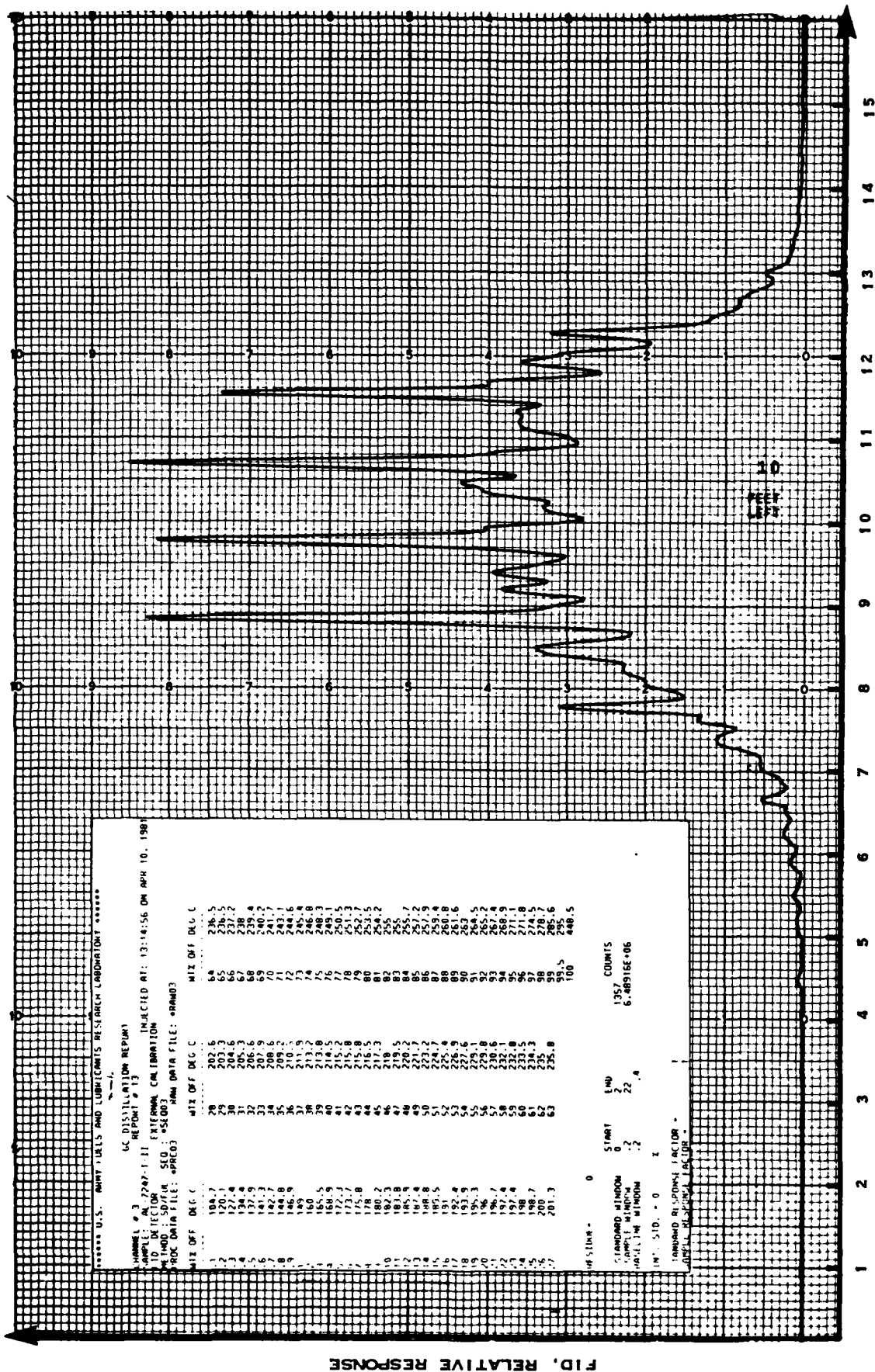
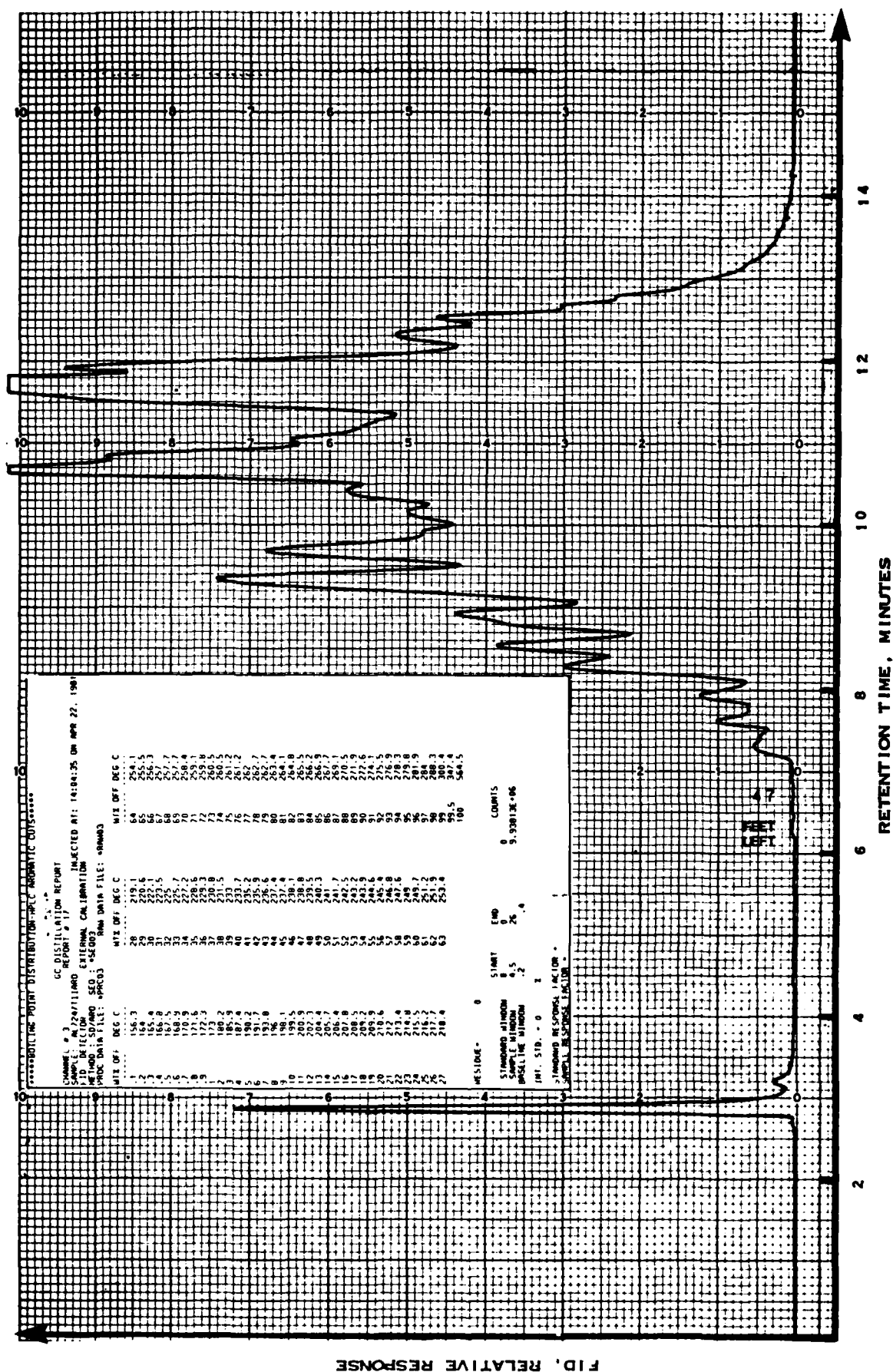


FIGURE 3. HPLC SEPARATION OF SATURATES AND AROMATIC/POLAR FRACTIONS OF PETROLEUM JP-5



RETENTION TIME, MINUTES

FIGURE 4. ASTM D 2887 BOILING POINT DISTRIBUTION OF PETROLEUM JP-5



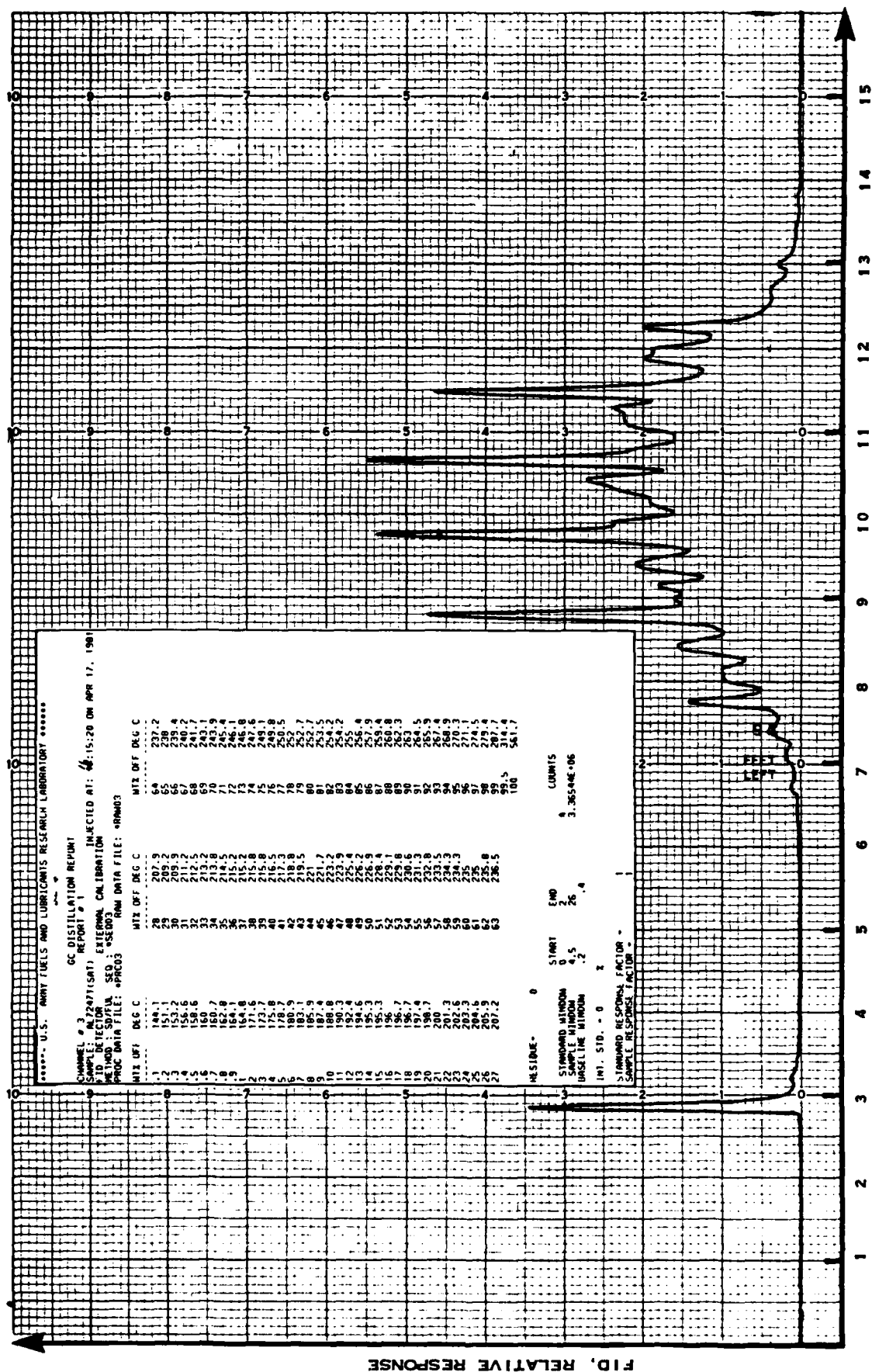


FIGURE 6. ASTM D 2887 BOILING POINT DISTRIBUTION
 OF SATURATE FRACTION FROM PETROLEUM JP-5

TABLE 3. OPERATING PARAMETERS
HEWLETT-PACKARD 5711 GAS CHROMATOGRAPH

Sample Size: 1 microliter
Flow Rate: 25 cc/min Helium

DETECTOR

Flame Ionization:
Temperature: 400°C
Hydrogen: 35 cc/min
Air: 360 cc/min
Helium (makeup): 25 cc/min

OVEN TEMPERATURE

Programmed: 0° to 390°C
Rate: 16°C/min
Initial Hold: 0 minutes
Final Hold: 4 minutes

DATA PROCESSING

Hewlett-Packard 3354 Laboratory Data System

COLUMN

6 ft x 1/8 inch SS, 5% SE-30 on Chromosorb G,
AW-DMCS, 80/100 mesh

In addition to the neat fuels, aliphatic and aromatic/polar fractions of each of the fuel samples were analyzed by high-resolution-capillary column GC, and their "fingerprint" patterns were obtained; examples of which are given in Figures 7, 8, and 9. The analyses were performed utilizing flame ionization (in Figures 7-9) and nitrogen-specific detectors. Table 4 gives the operating parameters for this process. The chromatographic data obtained were stored on data cartridges for future use in component identification and correlations.

Boiling point distribution is the predominant analytical method used in the characterization of a fuel. The ASTM D 2887 Boiling Point Distribution (BPD) method utilizes conventional, packed column, low-resolution GC. This method does not yield detailed component or element specific information.

TABLE 4. OPERATING PARAMETERS--
HEWLETT-PACKARD 5880A GAS CHROMATOGRAPH DUAL CAPILLARY COLUMNS--
HIGH RESOLUTION

Sample Size: 1 microliter
Split Ratio: 200:1
Flow Rate (Linear Velocity) 17.4 cm/sec Helium
Injector Temperature: 350°C

DETECTORS

Flame Ionization:
Temperature: 400°C
Hydrogen: 30 cc/min
Air: 350 cc/min
Helium (makeup): 25 cc/min

Nitrogen/Phosphorus:
Temperature: 400°C
Hydrogen: 3 cc/min
Air: 100 cc/min
Element Power:
100 (Zero=16)
Helium (makeup):
25 cc/min

OVEN TEMPERATURE

Programmed: 0° to 200°C
Rate 1: 4°/min
Initial Hold: 1 min
Final Hold: 0.1 min
Rate 2: 25°/min
Final Temperature: 320°
Final Hold: 15 min

DATA PROCESSING

H-P 5880 Lab Basic
Norm % Compensated Analysis

COLUMNS

1 and 2: SE-54, 50 meter x 0.3 mm ID Fused Silica Capillary Column

It would be advantageous if a D 2887 BPD could be obtained from a chromatographic analysis that was capable of yielding some component and/or element specific information at the same time. Earlier attempts to develop such a technique were unsuccessful because of chromatographic data storage limitations. This technique yields data for several hundred individual components in a fuel, and this data, together with the programming necessary for D 2887, exceeds the memory capacity of the Level 4 HP 5880 Gas Chromatograph.

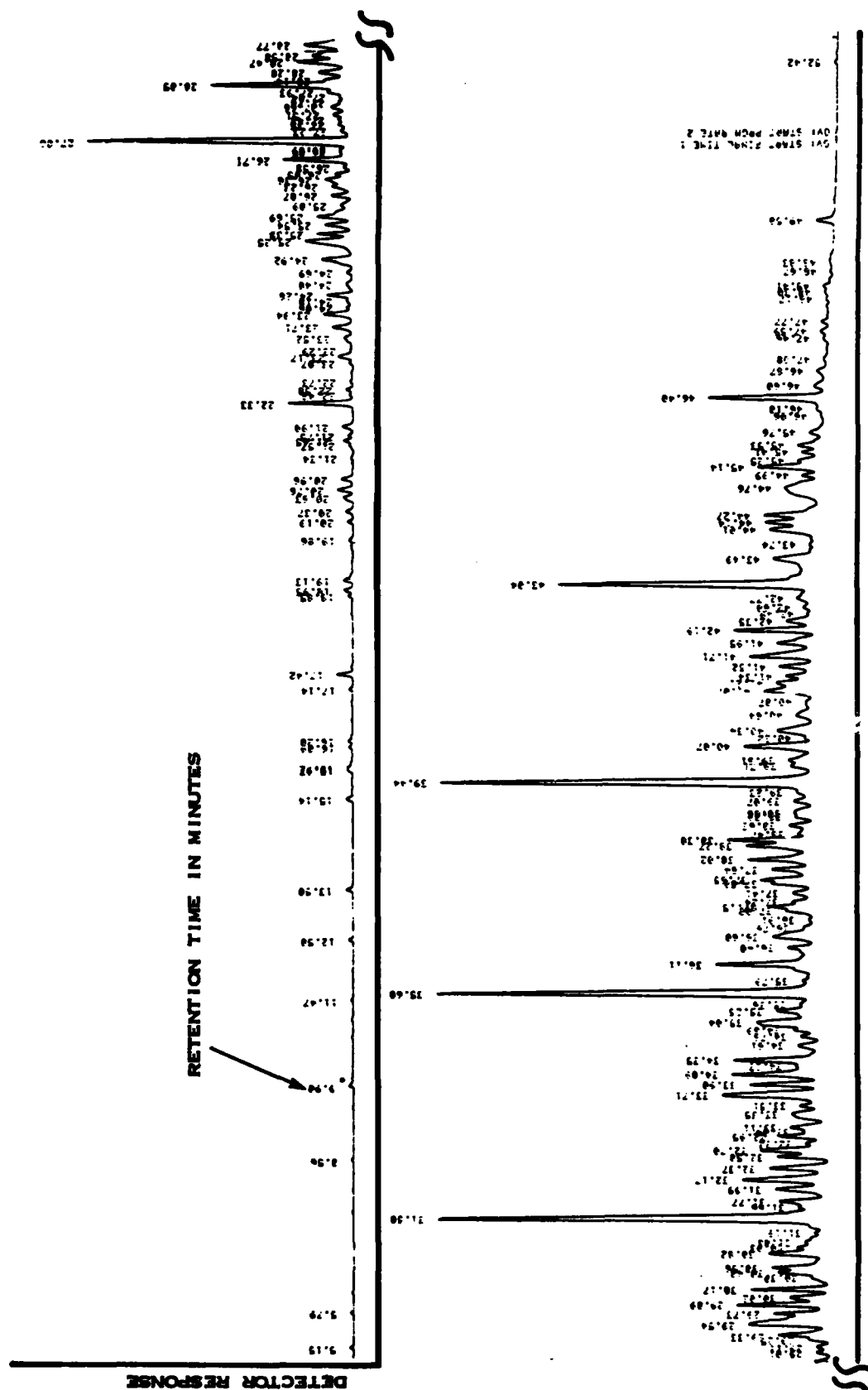


FIGURE 7. HIGH-RESOLUTION CAPILLARY COLUMN GC "FINGERPRINT" OF PETROLEUM JP-5

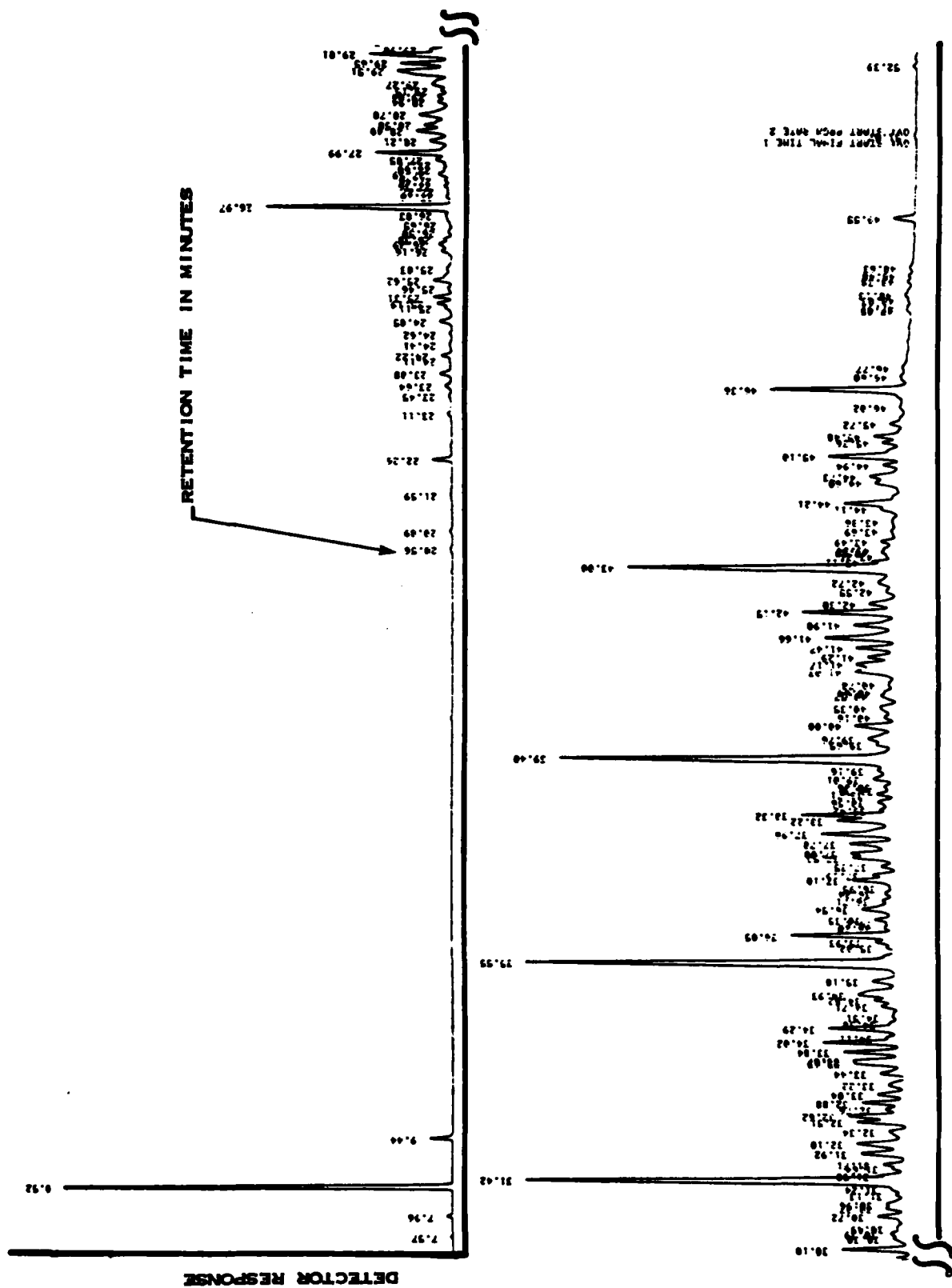
EAP 5833A MANUAL INJECTION 0 17:23 JUN 3 1981										31.77	347.61	VV	0.965	
AL-7247-T-11(PETROLEUM JP-5)										31.99	391.22	VV	1.086	
NORM % COMPENSATED ANALYSIS										32.17	644.16	VV	1.782	
RT	AREA	TYPE	CAL	AMOUNT	NAME					32.37	331.78	VP	1.059	
5.15	7.78	20	1	7.616E-05	N-C4					32.58	248.89	VP	0.689	
5.79	6.65	20		1.844E-02						32.78	439.96	VV	1.218	
8.56	5.22	20	4	1.065E-04	N-C6					32.82	124.99	VV	0.347	
9.50	22.32	20		6.194E-02						32.93	277.59	VV	0.765	
11.47	5.33	20		1.478E-02						33.04	72.04	VV	0.201	
12.50	14.28	20	6	1.057E-04	N-C7					33.11	242.01	VV	0.672	
13.50	25.18	20	19	6.966E-02	ETHPROPIONATE					33.35	378.14	VV	1.027	
15.14	27.75	20		7.780E-02						33.51	154.55	VV	0.429	
15.67	16.12	20		4.475E-02						33.71	911.49	VV	2.529	
15.72	17.94	VV	20	4.978E-02	TOLUENE					33.90	436.12	VV	1.210	PENTYLBENZ
16.09	11.53	20		3.195E-02						34.09	689.67	VV	1.914	
16.20	14.90	VV		4.134E-02						34.23	113.23	VV	0.314	
17.14	12.39	20		3.439E-02						34.35	669.35	VV	1.850	
17.42	59.71	20	7	3.678E-04	N-C8					34.61	218.16	VV	0.583	
18.85	17.60	20		4.804E-02						34.78	142.96	VV	0.397	
18.95	37.71	20		0.185						34.89	148.24	VV	0.411	
19.13	34.95	20		9.695E-02						35.04	665.90	VV	1.846	
19.66	14.46	20		4.012E-02						35.25	422.98	VV	1.176	
20.15	23.74	20		6.588E-02						35.36	171.20	VV	0.475	
20.37	40.48	VV	21	0.112	ETHYLBENZENE					35.60	2734.29	VV	11	3.659E-03 N-C12
20.63	65.46	VV		0.182						35.79	112.11	VV	0.314	
20.76	77.15	VV		0.214						36.11	942.77	VV	2.616	
20.96	61.71	VV		0.171						36.40	299.29	VV	0.831	
21.34	12.79	VV		5.549E-02						36.60	521.38	VV	1.447	
21.57	25.03	VV		6.947E-02						36.76	94.94	VV	0.263	
21.65	43.90	VV		0.122						36.91	63.89	VV	0.231	
21.75	22.39	VV		6.212E-02						37.39	185.91	VV	0.516	
21.90	62.98	20		0.175						37.15	299.17	VV	0.930	
22.32	279.81	VV	3	1.682E-03	N-C9					37.24	145.70	VV	0.404	
22.46	14.32	VV		3.974E-02						37.41	214.31	VV	2.595	
22.58	31.16	VV		6.672E-02						37.58	263.98	VV	0.733	
22.73	14.15	VV		7.927E-02						37.65	332.44	VV	0.923	
23.07	15.84	20		4.395E-02						37.84	342.58	VV	0.950	
23.17	32.19	VV		0.223						38.02	556.25	VV	1.544	
23.29	11.10	VV		3.088E-02						38.27	532.23	VV	1.477	
23.44	59.64	VV		0.180						38.38	478.93	VV	1.307	
23.71	113.05	VV		0.314						38.47	158.65	VV	0.418	
23.94	153.22	VV		0.425						38.63	282.37	VV	0.562	
24.06	23.01	VV		6.387E-02						38.79	109.66	VV	0.304	
24.16	27.37	VV		7.734E-02						38.86	223.37	VV	0.620	
24.28	114.58	VP		0.319						39.07	186.68	VV	0.518	
24.48	39.78	20		0.118						39.23	125.91	VV	0.349	
24.69	19.96	20		5.541E-02						39.44	2725.68	VV	25	7.565 N-C13
24.92	194.22	VV	22	0.539	N-PROPBENZ					39.71	197.14	VV	0.547	
25.25	334.88	VV		0.985						39.81	234.87	VV	0.652	
25.36	134.18	VV		0.372						40.07	534.82	VV	1.484	
25.54	129.61	VV		0.360						40.22	49.55	VV	0.138	
25.69	244.43	VV		0.678						40.34	389.90	VV	1.079	
25.89	59.76	VV		0.166						40.64	281.98	VV	0.561	
26.07	51.39	VV		0.226						40.87	54.40	VV	0.262	
26.23	96.77	VV		0.269						41.07	406.14	VV	1.127	
26.36	169.79	VV		0.471						41.21	221.47	VV	0.781	
26.45	58.51	VP		0.162						41.34	171.87	VV	0.475	
26.58	15.76	20		4.651E-02						41.52	205.24	VV	0.570	
26.71	266.26	20	23	0.729	1,2,4-MESENZ					41.71	491.93	VV	1.365	
26.85	6.53	VV		1.612E-02						41.95	272.42	VP	0.756	
26.89	10.14	VV		2.613E-02						42.19	399.11	VP	1.180	
27.06	1389.64	VV	9	5.687E-03	N-C10					42.35	174.78	VV	0.485	
27.23	30.27	VP		8.488E-02						42.49	8.92	VP	2.476E-02	
27.35	45.49	VV		0.126						42.68	24.74	VP	6.266E-02	
27.43	35.70	VV		0.118						42.77	73.57	VV	0.204	
27.51	79.08	VV		0.219						43.04	1561.98	VV	12	4.152E-03 N-C14
27.66	96.52	VV		0.268						43.49	299.23	SP	0.632	
27.74	35.54	VV		0.111						43.74	12.59	VP	3.521E-02	
27.84	26.96	VV		7.482E-02						44.31	338.93	VV	0.918	
27.93	35.03	VV		0.147						44.14	524.68	VV	0.901	
28.05	742.82	VV		2.062						44.27	375.05	VP	1.041	
28.14	52.52	VV		0.237						44.76	181.76	VV	0.500	
28.28	161.85	VV		0.449						44.99	59.52	VV	0.245	
28.47	157.15	VV		0.797						45.14	489.93	VV	1.175	
28.58	249.70	VV		0.691						45.25	135.17	VV	0.379	
28.77	31.31	VV		0.572						45.41	110.25	VV	0.780	
29.01	77.19	VV		0.195						45.53	175.35	VV	0.446	
29.11	74.54	VV		0.207						45.76	102.49	VP	0.284	
29.25	94.25	VV		0.253						46.06	15.59	VP	4.271E-02	
29.33	342.67	VV		0.952						46.16	47.73	VV	0.152	
29.54	865.17	VV		1.401						46.40	733.93	VV	0.877	
29.72	191.90	VV		0.792						46.68	102.42	VV	13	6.012E-04 N-C15
29.83	456.20	VV		1.294						46.97	36.53	VV	0.140	
30.02	281.21	VV		0.725						47.06	11.66	VV	7.653E-02	
30.17	367.93	VV		1.021						47.46	75.12	VV	0.181	
30.39	122.56	VV		0.340						47.59	38.19	VV	0.161	
30.49	299.06	VV		0.658						47.77	42.13	VV	0.174	
30.56	401.23	VV		1.115						48.17	29.59	VV	6.724E-02	
30.82	447.38	VV		1.241						48.30	46.24	VV	0.137	
30.93	187.71	VV		0.599						48.41	54.12	VV	0.150	
31.03	419.96	VV		0.910						48.67	11.72	VV	7.252E-02	
31.19	67.78	VV		0.181						48.83	46.76	VV	0.117	
31.52	71.379	VV	13	1.682E-02	N-C11					49.56	59.67	20	14	1.981E-04 N-C16
31.66	131.19	VV		0.254						52.42	11.36	20	15	6.27E-05 N-C17

FIGURE 7. HIGH-RESOLUTION CAPILLARY COLUMN GC "FINGERPRINT" OF PETROLEUM JP-5 (CONT'D)

EXP 5880A MANUAL INJECTION @ 14:04 JUN 12, 1981					36.36	340.85	VV	0.637
AL-7247-T-II (AROMATICS)					36.62	991.18	VV	1.852
NORM % COMPENSATED ANALYSIS					36.77	95.51	VP	0.178
RT	AREA	TYPE	CAL	NAME	37.04	793.66	PV	1.483
7.95	13.34	BB	2.493E-02		37.14	287.21	VV	0.537
8.51	2749.17	BB	4 3.785E-02	N-C6	37.34	742.14	VV	1.387
9.43	114.35	BB	0.214		37.61	688.30	VV	1.286
15.60	9.94	BB	20 1.857E-02	TOLUENE	37.74	112.01	VV	0.209
20.31	9.33	BB	21 1.744E-02	ETHYLBENZENE	37.92	561.24	VV	1.086
20.70	39.75	BB	5.372E-02		38.20	1307.25	VV	2.443
21.84	2.94	BB	4.108E-02		38.47	274.38	VV	0.513
22.26	0.20	BB	8 3.183E-05	N-C9	38.63	511.23	VV	0.956
23.40	12.67	BB	2.367E-02		38.84	729.15	VV	1.363
24.78	53.42	BB	22 9.984E-02	N-PROP BENZ	39.09	136.37	VP	0.256
25.17	190.69	BB	0.356		39.46	3504.02	VP	0.176
25.48	123.23	BP	0.240		39.71	458.68	VV	0.932
25.62	4.72	BP	8.824E-03		39.91	42.95	VV	0.027E-02
26.01	123.53	BP	0.231		40.12	1536.72	VV	2.872
26.29	10.89	VV	2.036E-02		40.32	1313.56	VV	2.455
26.65	495.06	BB	23 0.925	1,2,4-ME BENZ	40.64	299.05	VV	0.960
26.95	107.53	BP	9 1.996E-04	N-C10	40.76	39.42	VV	7.366E-02
27.29	36.44	BP	5.810E-02		40.87	127.83	VV	0.339
27.44	76.11	VP	0.142		41.04	769.21	VV	1.436
27.57	545.36	SV	1.625		41.20	271.40	VV	0.507
28.07	189.26	VV	3.204		41.42	153.03	VV	0.286
28.39	37.21	VV	6.953E-02		41.56	37.51	VV	7.010E-02
28.56	78.70	VV	0.147		41.75	394.82	VV	0.736
28.71	68.84	VP	0.129		42.01	261.47	BP	0.489
28.94	14.08	VP	2.632E-02		42.17	183.05	VP	0.193
29.20	201.22	VV	0.376		42.32	396.65	VV	0.742
29.28	479.52	VV	0.696		42.48	362.53	VV	0.565
29.48	613.67	VV	1.147		42.61	174.61	VV	0.327
29.61	523.23	VV	0.978		42.95	336.27	VV	0.529
29.75	137.13	VV	0.236		43.07	1069.51	VV	12 1.914E-03
29.82	54.47	VV	0.102		43.20	488.19	VV	0.763
29.96	508.65	VV	0.936		43.52	1806.30	VV	3.376
30.11	44.30	VV	8.278E-02		43.64	81.65	VV	0.153
30.44	529.90	VV	0.990		43.77	34.73	VV	6.496E-02
30.52	838.48	VV	1.552		43.86	48.19	VV	7.510E-02
30.80	437.10	VV	1.744		44.06	2026.77	VV	3.769
31.06	163.90	VV	0.344	NITROBENZ	44.20	1460.87	VV	2.730
31.28	54.91	VV	0.183		44.33	425.92	VB	0.796
31.38	327.72	VV	10 1.135E-03	N-C11	44.59	81.25	VB	0.152
31.48	324.10	VV	0.606		44.74	655.20	VV	1.0
31.61	512.31	VV	0.957		44.86	633.59	VV	1.
31.71	530.49	VV	0.991		45.20	176.52	VV	0.311
31.84	196.12	VV	0.367		45.12	57.77	VV	0.127
32.12	974.08	VV	1.822		45.26	523.11	VV	0.978
32.34	946.24	VP	1.768		45.40	77.95	VV	0.333
32.54	27.51	VP	5.140E-02		45.59	127.80	VV	0.310
32.63	165.98	VV	0.310		45.72	11.91	VV	0.160
32.72	52.71	VV	9.858E-02		45.78	124.23	VP	0.232
32.99	544.89	VV	1.017		45.96	38.35	VP	7.167E-02
33.09	370.66	VV	0.506		46.18	297.38	VP	0.556
33.35	1229.74	VV	2.298		46.35	349.90	VV	13 1.333E-03
33.69	1659.77	VV	3.476		46.58	592.07	VV	1.186
33.85	573.39	VV	1.072	PENTYLBENZ	46.89	408.70	VV	0.764
34.00	506.85	VV	0.951		47.08	289.98	VV	0.542
34.09	514.89	VV	0.962		47.45	310.41	VV	0.580
34.26	462.63	VV	0.865		47.60	436.43	VV	0.816
34.28	800.39	VV	1.496		47.78	387.56	VV	0.724
34.58	526.26	VV	0.987		48.02	139.62	VV	0.251
34.73	40.06	VV	7.487E-02		48.32	466.19	VP	0.909
35.04	265.36	VV	2.365		48.85	152.72	VP	0.261
35.12	343.56	VV	0.642		48.93	92.68	VB	0.173
35.21	534.71	VV	0.999		49.44	45.72	VB	0.545E-02
35.33	667.99	VV	1.248		49.53	51.64	VV	9.650E-02
35.52	1402.48	VV	11 1.264E-03	N-C12	49.66	73.01	VV	14 1.443E-04
35.76	452.02	VV	0.845		49.93	51.50	VV	0.115
35.80	427.64	VV	0.799		50.66	18.65	VP	3.491E-02
36.16	461.11	VV	0.862		59.32	44.34	BB	17 9.042E-04
36.25	210.91	VV	3.394					

MULTIPLIER = 1

FIGURE 8. HIGH-RESOLUTION CAPILLARY COLUMN GC "FINGERPRINT" OF AROMATICS/POLAR FRACTION OF PETROLEUM JP-5 (CONT'D)



E-2 5000A MANUAL INJECTION @ 14:34 JUN 10, 1981				34.30	24.26	VP	0.103	
AL-7247-T-11(SAT)				34.51	6.40	PO	2.741E-02	
NORM % COMPENSATED ANALYSIS				34.71	70.16	OV	0.331	
RT	AREA	TYPE	CAL	AMOUNT	NAME			
7.37	5.16	BB		2.192E-02				
7.96	16.01	BB		6.777E-02				
8.32	2236.68	BB	4	6.977E-02	N-C6			
9.44	74.69	BB		0.316				
20.36	2.35	BB	21	9.927E-03	ETHYLBENZENE			
20.89	8.64	BB		3.650E-02				
21.59	9.50	VV		3.631E-02				
22.26	61.22	BP	8	7.140E-04	N-C9			
23.11	16.01	PV		7.117E-02				
23.45	9.32	BP		3.946E-02				
23.64	30.04	PO		0.131				
23.88	56.21	OV		0.230				
24.11	10.00	VV		4.235E-02				
24.22	44.93	VP		0.190				
24.41	13.71	PO		5.805E-02				
24.52	7.00	BB		2.964E-02				
24.85	71.13	OV	22	0.301	N-PROPENEZ			
25.11	40.59	VV		0.172				
25.19	66.95	VV		0.203				
25.31	79.58	VV		0.320				
25.46	25.76	VV		0.109				
25.62	137.76	VV		0.566				
25.93	39.16	VV		0.166				
26.16	53.24	OV		0.247				
26.29	27.08	VV		0.369				
26.38	33.97	VV		0.144				
26.50	19.12	VP		8.094E-02				
26.65	2.21	PO	23	9.350E-03	1,2,4-MEBENZ			
26.83	4.05	BP		1.716E-02				
26.97	862.20	PO	9	3.622E-03	N-C10			
27.16	19.54	PV		8.270E-02				
27.27	12.46	VV		5.275E-02				
27.36	27.40	VV		0.116				
27.46	26.46	VV		2.112				
27.59	68.38	VV		0.209				
27.68	30.36	VV		0.129				
27.77	22.66	VV		9.593E-02				
27.85	12.95	VV		0.351				
27.99	375.51	VV		1.590				
28.21	122.73	VV		0.520				
29.40	195.77	VV		0.829				
28.50	100.16	VV		0.703				
28.70	233.66	VV		0.989				
28.94	51.66	VV		0.219				
29.04	59.94	VV		0.250				
29.15	12.11	VV		0.136				
29.27	139.76	VV		0.592				
29.51	391.26	VV		1.656				
29.65	215.75	VV		0.930				
29.81	387.07	VV		1.623				
29.90	79.74	VV		0.330				
30.10	322.46	VV		1.365				
30.30	108.12	VV		0.450				
30.39	132.93	VV		0.563				
30.49	35.75	VV		0.363				
30.72	230.34	VV		0.975				
30.87	117.64	VV		0.490				
30.96	172.72	VV		0.771				
31.13	67.11	VV	30	0.204	NITROBENZ			
31.24	66.23	VV		0.200				
31.42	2635.20	VV	10	2.908E-02	N-C11			
31.50	89.13	VV		0.377				
31.64	79.71	VV		0.337				
31.71	120.91	VV		0.512				
31.92	310.56	VV		1.349				
32.10	403.04	VV		1.706				
32.34	164.72	VV		0.697				
32.51	260.00	VV		1.101				
32.62	370.67	VV		1.604				
32.76	137.84	VV		0.503				
32.80	240.91	VV		1.054				
33.04	139.36	VV		0.602				
33.22	150.55	VP		0.637				
33.44	169.37	PV		0.717				
33.63	234.79	VV		0.994				
33.67	229.75	VV		0.973				
33.64	308.73	VV	24	1.307	PENTYLBENZ			
34.02	307.67	VV		1.641				
34.11	52.91	VV		0.224				
34.29	309.94	VV		1.650				
				34.30	24.26	VP	0.103	
				34.51	6.40	PO	2.741E-02	
				34.71	70.16	OV	0.331	
				34.82	117.02	VV	0.495	
				34.93	354.51	VV	1.501	
				35.10	162.46	VV	0.600	
				35.55	2593.05	VV	11.529E-03	N-C12
				35.82	42.91	VV	0.102	
				35.93	115.53	VV	0.409	
				36.05	570.16	VV	2.447	
				36.20	66.90	VV	0.204	
				36.27	31.72	VV	0.134	
				36.35	120.32	VV	0.543	
				36.54	200.00	VV	1.223	
				36.71	74.01	VV	0.313	
				36.84	72.60	VV	0.306	
				36.95	22.33	VV	9.936E-02	
				37.10	272.41	VV	1.153	
				37.19	110.32	VV	0.467	
				37.35	70.20	VV	0.323	
				37.52	249.34	VV	1.056	
				37.60	251.76	VV	1.066	
				37.76	305.16	VV	1.292	
				37.96	529.63	VV	2.242	
				38.22	330.50	VV	1.435	
				38.32	400.14	VV	2.066	
				38.42	73.77	VV	0.312	
				38.56	152.05	VV	0.644	
				38.71	107.71	VV	0.456	
				38.79	13.57	VV	5.743E-02	
				38.86	73.75	VV	0.312	
				39.01	140.10	VV	0.763	
				39.16	124.62	VV	0.528	
				39.40	2463.51	VV	25.104E-03	N-C13
				39.65	114.77	VV	0.406	
				39.76	265.02	VV	1.122	
				40.00	203.00	VV	1.201	
				40.16	71.93	VV	0.304	
				40.35	150.93	VP	0.673	
				40.57	105.53	PV	0.440	
				40.64	54.02	VV	0.229	
				40.73	66.94	VV	0.283	
				41.03	330.41	VV	1.433	
				41.17	200.50	VV	1.222	
				41.29	157.01	VV	0.605	
				41.47	217.56	VV	0.923	
				41.66	495.63	VV	2.099	
				41.80	204.56	VV	1.205	
				42.15	513.62	VV	2.175	
				42.30	191.77	VP	0.812	
				42.55	30.20	PO	0.126	
				42.72	106.84	OV	0.452	
				43.00	1776.92	VV	12.720E-03	N-C14
				43.11	64.93	VV	0.360	
				43.21	24.34	VV	0.103	
				43.29	65.05	VV	0.270	
				43.34	54.51	VV	0.231	
				43.49	60.26	VP	0.269	
				43.69	20.03	PV	0.119	
				43.86	41.01	VP	0.176	
				44.11	57.50	PV	0.413	
				44.21	351.74	VV	1.409	
				44.60	195.34	PV	0.827	
				44.73	225.50	VV	0.355	
				44.94	94.05	VV	0.390	
				45.10	354.72	VV	2.340	
				45.36	103.57	VV	0.430	
				45.44	191.56	VV	0.811	
				45.72	99.06	OV	0.423	
				46.02	11.93	BB	5.032E-02	
				46.36	790.00	BP	13.745E-03	N-C15
				46.60	50.01	PV	0.214	
				46.77	34.64	OV	0.147	
				47.05	21.03	PV	8.302E-02	
				47.91	16.01	VV	7.115E-02	
				48.03	17.00	VV	7.570E-02	
				48.13	45.22	VV	0.191	
				48.30	25.61	VV	0.100	
				48.40	19.69	VV	8.334E-02	
				48.63	10.55	OV	7.067E-02	
				49.55	113.10	BB	14.506E-04	N-C16
				51.07	9.79	BB	4.146E-02	
				52.39	13.71	BB	15.145E-04	N-C17
				MULTIPLIER = 1				

A medium resolution chromatographic analysis was successfully developed. The operating parameters are shown in Table 5. The BPD reported by this technique is in close agreement with the BPD reported by the conventional D 2887 technique (Figures 10 and 10a, respectively). Figure 11 graphically illustrates the close agreement between the conventional and medium resolution D 2887 techniques. In addition, it can yield component and/or element specific information, however, not quite to the extent of a high-resolution analysis (as in Table 4). Note that the GC Distillation Report in Figure 10 is based on Boiling-Point Distribution by Gas Chromatography (BPDGC) software upgrade as presented in an earlier report.(25)

TABLE 5. OPERATING PARAMETERS--
HEWLETT-PACKARD 5880A GAS CHROMATOGRAPH DUAL CAPILLARY COLUMN--
MEDIUM RESOLUTION

Sample Size: 1 microliter
Split Ratio: 200:1
Flow Rate (Linear Velocity) Helium 17.4 cm/sec
Injector Temp: 350°C

DETECTORS

Flame Ionization:
Temperature: 400°C
Hydrogen: 30 cc/min
Air: 350 cc/min
Helium (makeup): 25 cc/min

Nitrogen/Phosphorus:
Temperature: 400°C
Hydrogen: 3 cc/min
Air: 100 cc/min
Element Power:
100 (Zero=16)

OVEN TEMPERATURE

Programmed: 0° to 320°C
Rate: 20° C/min
Initial Hold: 0 min
Final Hold: 14 min
Start of Integration: 4.5 minutes

DATA PROCESSING

Figure 10a--H-P 5880 Lab Basic
Simulated Distillation-D 2887 Software
Figure 10--H-P-3354 Laboratory Data System

COLUMNS

1 and 2: SE-54; 50 meter x 0.3mm ID Fused Silica
Capillary Column

GC DISTILLATION REPORT
 CHANNEL # 3 REPORT # 13
 SAMPLE: AL-7247-T-II INJECTED AT: 13:14:56 ON APR 10, 1981
 FID DETECTOR EXTERNAL CALIBRATION
 METHOD : SD/FUL SEQ : *SEQ03
 PROC DATA FILE: *PRC03 RAW DATA FILE: *RAW03

WT% OFF	DEG C	WT% OFF	DEG C	WT% OFF	DEG C
.1	104.7	28	202.6	64	236.5
.2	120.1	29	203.3	65	236.5
.3	127.4	30	204.6	66	237.2
.4	134.4	31	205.3	67	238
.5	137.9	32	206.6	68	239.4
.6	141.3	33	207.9	69	240.2
.7	142.7	34	208.6	70	241.7
.8	144.8	35	209.2	71	243.1
.9	146.9	36	210.5	72	244.6
1	149	37	211.9	73	245.4
2	160	38	213.2	74	246.8
3	165.5	39	213.8	75	248.3
4	168.9	40	214.5	76	249.1
5	172.3	41	215.2	77	250.5
6	173.7	42	215.8	78	251.3
7	175.8	43	215.8	79	252.7
8	178	44	216.5	80	253.5
9	180.2	45	217.3	81	254.2
10	182.3	46	218	82	255
11	183.8	47	219.5	83	255
12	185.9	48	220.2	84	255.7
13	187.4	49	221.7	85	257.2
14	188.8	50	223.2	86	257.9
15	189.5	51	224.7	87	259.4
16	191	52	225.4	88	260.8
17	192.4	53	226.9	89	261.6
18	193.9	54	227.6	90	263
19	195.3	55	229.1	91	264.5
20	196	56	229.8	92	265.2
21	196.7	57	230.6	93	267.4
22	197.4	58	232.1	94	268.9
23	197.4	59	232.8	95	271.1
24	198	60	233.5	96	271.8
25	198.7	61	234.3	97	274.5
26	200	62	235	98	278.7
27	201.3	63	235.8	99	285.6
				99.5	295
				100	448.5

RESIDUE= 0

	START	END	COUNTS
STANDARD WINDOW	0	2	1357
SAMPLE WINDOW	.2	22	6.48916E+06
BASELINE WINDOW	.2	.4	

INT. STD. = 0 %

STANDARD RESPONSE FACTOR = 1
 SAMPLE RESPONSE FACTOR = 1

FIGURE 10. D 2887 GC DISTILLATION REPORT BY
 CONVENTIONAL SIMULATED DISTILLATION

ASTM D-2887 REPORT

SAMPLE: AL-7247-T(CH1)12/22

MANUAL INJECTION @ 13:20 DEC 22, 1981

% OFF	DEG C	% OFF	DEG C
----	----	----	----
IBP	132		
1	140	51	226
2	152	52	227
3	159	53	228
4	164	54	229
5	171	55	230
6	174	56	231
7	175	57	232
8	176	58	233
9	177	59	234
10	178	60	236
11	179	61	237
12	181	62	238
13	182	63	238
14	183	64	239
15	184	65	239
16	186	66	240
17	187	67	241
18	188	68	242
19	190	69	243
20	192	70	244
21	193	71	245
22	194	72	246
23	194	73	247
24	195	74	248
25	196	75	249
26	197	76	250
27	198	77	251
28	199	78	252
29	201	79	253
30	202	80	254
31	204	81	255
32	205	82	256
33	207	83	257
34	208	84	258
35	209	85	259
36	210	86	261
37	211	87	263
38	213	88	264
39	215	89	265
40	215	90	266
41	216	91	268
42	217	92	269
43	217	93	271
44	218	94	273
45	219	95	275
46	220	96	277
47	221	97	280
48	222	98	284
49	222	99	289
50	224	FEP	295

FIGURE 10a. D 2887 GC DISTILLATION REPORT BY
MODIFIED SIMULATED DISTILLATION

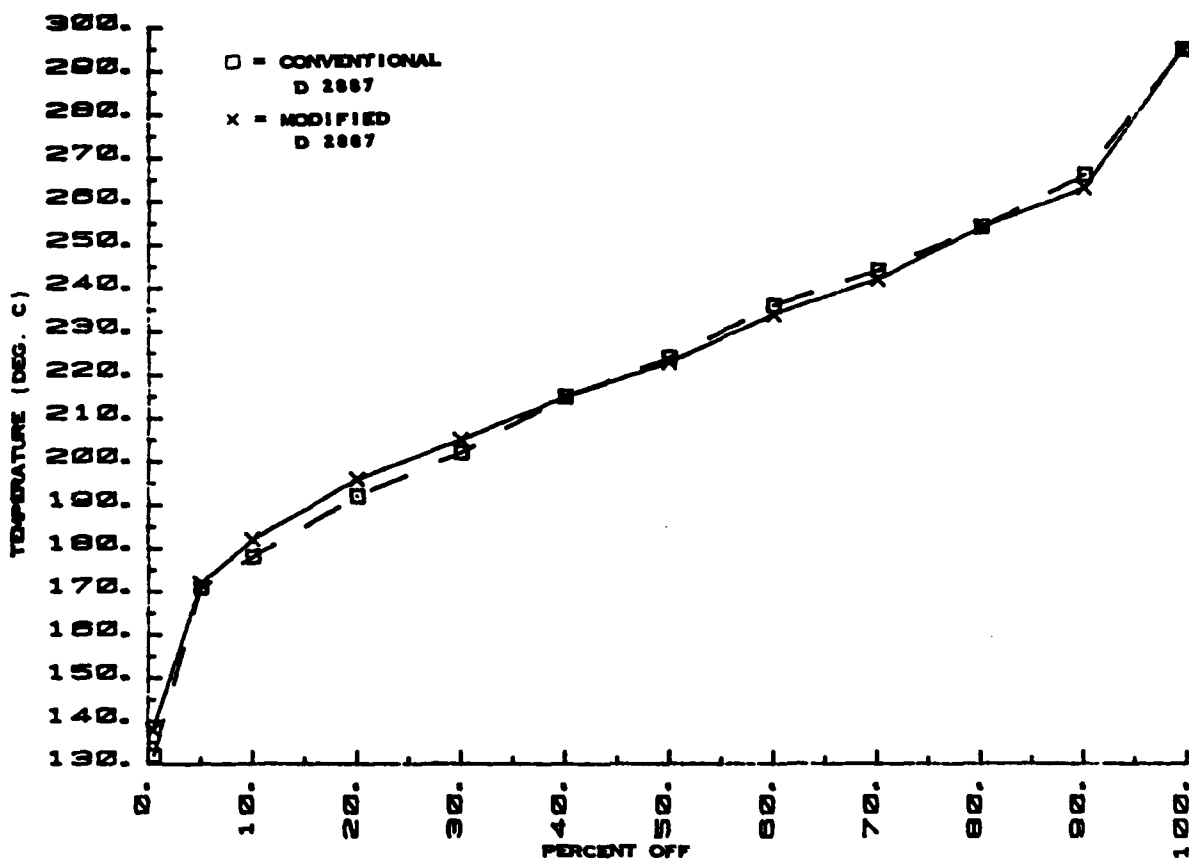


FIGURE 11. COMPARISON OF CONVENTIONAL VS. MODIFIED D 2887
BOILING POINT DATA

For ease in comparing the standard ASTM D 2887 and two modified D 2887 capillary column techniques (Tables 4 and 5), Figure 12 provides a plot of Kovats Indices (I_R) versus retention time for the n-saturates.(26) The conditions in Table 5 appear to provide peak resolution comparable to, but somewhat improved over those currently in use at the Aero Propulsion Laboratory where similar fuels are being investigated (27).

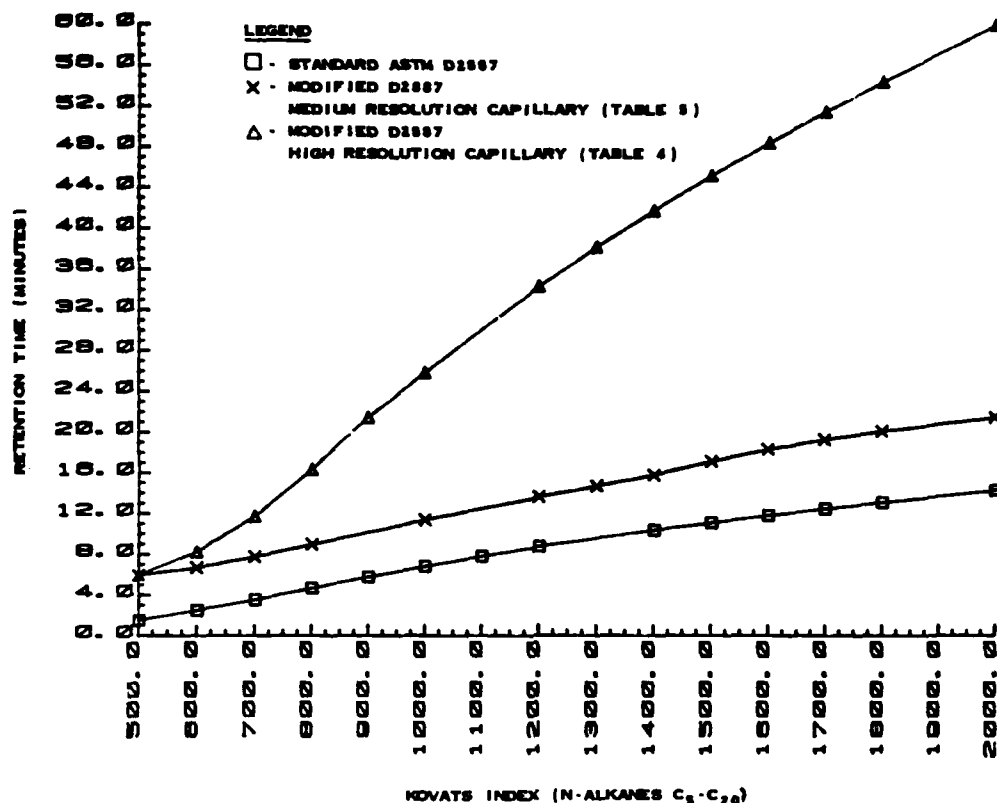


FIGURE 12. COMPARISON OF ABSOLUTE RETENTION TIME AND KOVATS INDICES FOR VARIOUS CHROMATOGRAPHIC METHODS

In addition to calculating the ASTM D 2887 BPD, the program is capable of reporting the correlation to ASTM D 86, "Distillation of Petroleum Products" (wide range) or ASTM D 1160, "Distillation of Petroleum Products at Reduced Pressure" (Table 6).

Equations for calculating the correlation of the modified D 2887 BPD to Reid Vapor Pressure (RVP) have been developed (28), but require some refining to bring the results into agreement with experimental results (Table 6). Other correlations have been developed equating the D 2887 BPD data to °API gravity, flash point, and viscosity.(4) However, the application of these correlation equations requires some additional refinement of the constants and selected data points to allow their use.

TABLE 6. D 86 AND REID VAPOR PRESSURE CORRELATION RESULTS

ASTM CORRELATION FOR D 2887

Sample: AL-7247-T(CH1)12/22

<u>% OFF</u>	<u>D 86 CORRELATION</u>		<u>D 86, actual</u>
	<u>DEG C (^oF)</u>		<u>DEG F</u>
IBP	174	(345)	355
10	194	(381)	391
20	201	(394)	401
30	207	(405)	407
50	219	(426)	425
70	232	(450)	445
90	239	(462)	475
95	247	(477)	485
FBP	265	(509)	511

Reid Vapor Pressure Correlation for AL-7247-T(CH1)12/22 = 2.87

Analysis by proton (¹H) and carbon-13 (¹³C) nuclear magnetic resonance (NMR) was explored to determine if NMR can be a useful technique in the characterization of fuels. Samples of the eight test fuels were sent to a commercial laboratory for this analysis. Interpretation of the spectral data along with UV aromaticity, BPD for carbon number range and molecular weight were expected to yield percent CH₃, CH₂, CH, aromatic carbon, amount of substitution on aromatic rings, number of fused rings, length of side chains, and degree of branching. Since no standard data requirements existed, considerable effort was expended in establishing the most dependable and significant values to report as shown in Table 7.(29) However, examination of the NMR analytical data of the eight test fuels showed significant differences compared to the results of the standard analytical procedures for aromatic and aliphatic compounds in Table 8, and no immediate benefit could be derived from the use of this technique.

TABLE 7. MOLE PERCENT CARBON AND HYDROCARBON DISTRIBUTIONS USING NMR

Sample	% CARBON DISTRIBUTION				% HYDROGEN DISTRIBUTION							Average n-Paraffin Chain Length (N C)	$\frac{\text{CH}_3}{\text{CH}_2}$	
	Gross		Subdivided											
	Aromatic	Aliphatic	Protonated Aromatic	Substituted Aromatic	n-Paraffin	Cyclo Paraffins	Aromatic	Aliphatic	$\frac{\text{CH}_2}{\text{CH}_3}$	$\sigma(1)$				
8436-F	14.1	85.9	8.0	6.1	35.1	50.8	3.7	96.3	8.8	54.7	32.7	0.43	10.2	0.60
8437-F	15.5	84.5	7.8	7.6	33.9	50.6	3.9	96.1	8.0	55.8	32.1	0.49	12.5	0.58
8907-F	6.6	93.4	3.6	3.0	39.0	54.4	2.6	97.4	5.1	53.5	37.6	0.46	8.8	0.70
9089-SP	15.3	84.7	8.2	7.0	37.0	47.1	3.9	96.1	8.1	54.0	33.9	0.46	10.0	0.63
9847-SP-T	10.2	89.8	5.7	4.7	32.9	56.9	2.6	97.4	5.1	53.5	37.6	0.46	8.8	0.70
7247-T	13.0	87.0	7.4	5.6	28.7	58.3	3.9	96.1	8.4	54.2	33.4	0.43	9.8	0.64
6354-T	9.6	90.4	5.2	4.4	22.9	67.5	2.7	97.3	7.1	52.5	37.7	0.46	9.7	0.72
6526-T ⁽²⁾	15.3	84.7	8.9	6.4	49.3	35.4	4.4	95.2	7.6	55.4	30.5	0.42	10.8	0.55

(1) σ ~ That fraction of aromatic edge atoms which are substituted.

(2) This sample contains some olefins (0.4% olefinic hydrogen distribution).

TABLE 8. COMPARISON OF NMR, FIA, AND HPLC ANALYSES
FOR ALIPHATICS AND AROMATICS DISTRIBUTION

	<u>8436-F</u>	<u>8437-F</u>	<u>8907-F</u>	<u>9089-SP</u>	<u>9847-SP-T</u>	<u>7247-T</u>	<u>6354-T</u>	<u>6526-T</u>
<u>NMR</u> ⁽¹⁾								
Aliphatics	85.9	84.5	93.4	84.7	89.8	87.0	90.4	84.7
Aromatics	14.1	15.5	6.6	15.3	10.2	13.0	9.6	15.3
<u>UV</u> ⁽¹⁾								
Mono	13.5	11.6	5.1	13.8	5.8	9.8	7.0	15.1
Di	1.4	4.0	0.9	1.2	0.7	2.2	0.1	1.7
Tri	0.00	0.05	0.03	0.00	0.03	0.01	0.01	0.02
<u>FIA*, VOL%</u>								
Aliphatics	79	70	76	79	90	79	82	76
Aromatics	22	30	24	21	10	21	18	24
<u>HPLC, WT%</u>								
Aliphatics	75	72	77	76	ND	81	85	77
Aromatics**	25	28	23	24	ND	19	16	23

(1) Wt% Aromatic Ring Carbon; net volume of aromatic hydrocarbon, see Reference 23.

* Fluorescent Indicator Absorption (ASTM D 1319).

** Includes Olefins.

Table 9 lists the chemical/physical properties initially explored for compatibility with correlative methods. These properties rank high in importance in determining a fuel's properties and have been the subject of considerable exploratory correlative work. (4)

The problem of analyses comparison is frequently encountered in gas and liquid chromatography, e.g., when a comparison of the analytical results of

TABLE 9. CHEMICAL/PHYSICAL PROPERTIES:
INITIAL CANDIDATES FOR CORRELATION TECHNIQUES
UTILIZING D 2887 DATA

- D 86 Correlation
 - Reid Vapor Pressure Correlation
 - Degrees API Correlation
 - Flash Point Correlation
 - Viscosity Correlation
 - Freezing Point Correlation
-

the fractions of a chromatographic run is required, or a survey of the concentration distribution of a chemical substance over several samples would be advantageous. In these and other fields of application, a visual display of the results can be a useful aid when drawing conclusions concerning the chemical differences between similar samples with differing physical parameters.

In order to obtain this type of data, the Hewlett-Packard 3354B/C Laboratory Data System was updated to allow the use of graphics terminals for obtaining "profiled" plots of multiple chromatographic analyses. The necessary programming was entered and stored in the computer memory. A graphics CRT terminal and printer were utilized to test the profiling capability. The results were conditionally successful. Because of the large size of the program, it must be divided into several programs, all linked together. Figure 13 is an example of the results which can be achieved when four chromatograms (in this case for four different JP-5 samples) are profiled for comparison.

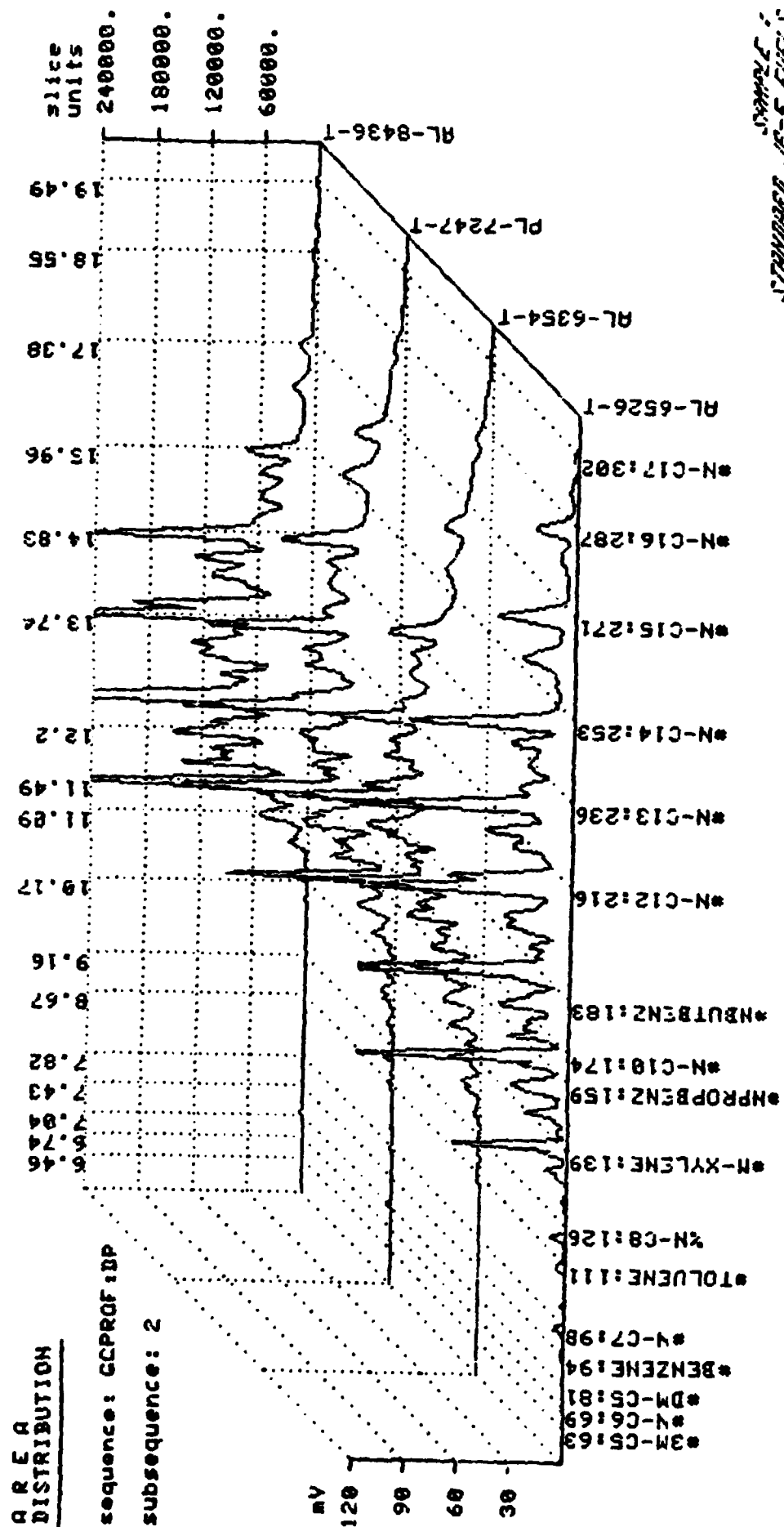


FIGURE 13 PROFILED PLOT OF MODIFIED D 2887 SIMULATED
DISTILLATION OF FOUR JP-5 FUELS

V. CURRENT STATUS

As stated previously, boiling point distribution by gas chromatography in a sophisticated mode appears to be the method of choice in establishing a number of correlations to individually determined experimental values.

The modified ASTM D 2887 method employing capillary columns yields BPD data consistent with the conventional packed column method. In addition, it is capable of giving component identification data and data to perform mathematical correlations. The equations for calculating the correlation to RVP, D 86, D 1160, °API gravity, flash point, and viscosity have been determined. However, in some cases, the constants and/or the data points used need adjustment to bring the results into closer agreement with the experimentally derived results.

Initial attempts to generate a "profiled" gas chromatogram of several similar fuels from different sources using graphics computer terminals has been successful. However, the programming requires some additional refining.

A gas chromatographic method for performing detailed component analysis, or "fingerprinting", of fuels utilizing fused silica capillary columns, temperature programming with subambient temperature capability, and selective detectors has been developed. Initial work has been performed with a flame ionization detector (FID) for a complete hydrocarbon presentation and some limited work with a nitrogen-phosphorus sensitive detector (NPD) to look at only those components containing nitrogen.

More than 40 compound identifications have been made with the FID system. To date, no work has been initiated toward identifying the nitrogen-containing compounds. Additionally, while a UV specific detector has been installed, no work has been done to demonstrate the specificity of this detector for aromatics. The need for a sulfur detector has been identified, but has yet to be purchased, installed, or demonstrated.

VI. CONCLUSIONS AND RECOMMENDATIONS

Development of a modified D 2887 boiling point distribution method to yield

component specific identification in addition to BPD consistent with the conventional D 2887 method has been successful. The modified D 2887 uses fused-silica capillary columns and will be capable of supplying the data necessary to calculate automatically correlations to RVP, ASTM D 86, D 1160, °API gravity, flash point, and viscosity already under investigation. These correlations, together with the method for "fingerprint" or more detailed chromatographic analysis of fuels, other correlations such as freezing point, and the capability to "profile" several chromatograms for direct visual comparison, can prove to be a very powerful tool in the characterization of candidate fuels with a minimal amount of laboratory analytical testing, thus effectively complementing the testing of candidate fuels both in the laboratory and by actual engine testing.

With the exception of the modified ASTM D 2887 boiling point distribution method discussed earlier, most of the work reported is in the initial stages of development. The sophisticated gas chromatographic instrumentation necessary for the approach taken was delivered in mid-March 1981 and required installation and check-out. The graphics computer terminals and computer programs necessary for profiling were not available until late September 1981, and are not dedicated to the GC system.

Because the key to the development of correlations and profiling was a form of ASTM D 2887, which would yield acceptable BPD data and the capability of component identification, emphasis was placed on the development of a modified ASTM D 2887 method and the development of a gas chromatographic method for obtaining detailed "fingerprints" of fuels. Work on the development of correlations and profiling did not start until September 1981.

The indications are that the development of correlations from BPD data is a viable and practical approach. Therefore, it is recommended that:

- (1) Efforts to refine the correlation equations already developed for RVP, °API, flash point, and viscosity to bring them into closer agreement with experimental results should be continued, but with an expanded set of sample fuels covering a broader boiling point range. This expanded set of sample fuels should be sufficiently large to establish a fuel

identification approach similar to that in Reference 30.

- (2) The development of correlations for other characteristic parameters such as freezing point should be investigated.
- (3) A quantitative sample introduction approach should be investigated which may allow external calibration for calculation of heat of combustion, noncombustible components, etc.
- (4) The development of the use of computer-assisted chromatogram profiling should be continued to include dedicated use of graphics terminals, both CRT type and plotting type. This would allow the generation of this type of reporting data to be effectively utilized.
- (5) The modified ASTM D 2887 method should be expanded and demonstrated using property specific detectors to include nitrogen, sulfur, and the aromatic sensitive UV detector. Thermal conductivity detectors should be evaluated as to their ability to provide volumetric rather than gravimetric data as does the FID for lower boiling materials.
- (6) The use of other instrumentation such as GC/MS, HPLC, IR, and UV/VIS, and NMR should be explored further to determine what additional contributions each might be able to make toward the expanded/rapid characterization of fuels.
- (7) Expand collaborative effort with other Federal/Military Laboratories developing glass capillary gas chromatographic methods for characterizing military fuels.
- (8) Expand analytical approach, where necessary, to assure program is in concert with and applicable to the emerging AIRLAND 2000 Battle Concept (31), which places emphasis on use of other than just conventional fossil fuels.

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FORT BELVOIR VA 22060

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ATTN ATSH-CD-MS-M
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